

PATENT SPECIFICATION

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(19)



(54) CHEMICAL MONITORING DEVICES

- (71) We, KODAK LIMITED, a Company registered under the law of England, of Kodak House, Station Road, Hemel Hempstead, Hertfordshire, do hereby declare the invention for which pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:
- This invention relates to gas-sensitive coatings containing a concentrated acid intended for use in chemical monitoring applications.
- Chemical monitoring or analytical devices are known which comprise tubes containing granules through which a known volume of gas is drawn. The extent and colour of the change effected on the contents of the tube is indicative of the quality and identity of gas(es) respectively in the sample analysed. A large number of such tubes are available many of which contain a concentrated acid. The best known of these are perhaps the variety which are sensitive to alcohol (ethanol) and may be used for breath-testing.
- According to the present invention there is provided a chemical monitoring device containing a concentrated acid which comprises a support having coated thereon a layer comprising a concentrated acid adsorbed onto particles of an inert adsorbent, a colour-forming reagent and a binder comprising a non-degradable synthetic polymer.
- The inert adsorbent is preferably alumina, Kieselguhr, titanium dioxide or silica gel. Charcoal could also be used in certain circumstances, for example if the layer was coated over a white titanium dioxide-containing layer for viewing through the base, or under a titanium dioxide layer for viewing from above. The particles of the adsorbent preferably have an average size of from 1-100, preferably 3-30 microns.
- The concentrated acid may be inorganic or organic, for example, concentrated sulphuric, nitric or phosphoric acid, glacial acetic acid or saturated aqueous citric acid.
- The colour-forming reagent may, for example, be a water-soluble dichromate (for the detection of ethanol); iodine pentoxide (for the detection of toluene); benzene (for the detection of formaldehyde); or formaldehyde or a compound capable of releasing formaldehyde (for the detection of xylene).
- The binder is a synthetic polymer which is not degraded by the concentrated acid. It is preferably a polyacrylamide homopolymer or copolymer. It has been found that the purity of the polymer is significant as it is the impurities that tend to degrade in the presence of the concentrated acid. High purity polymers are therefore preferred.
- Upon exposure of the coating of the appropriate gas, a visible dye is formed. The optical density of the dye is proportional to the concentration of the reducing gas in its proximity and the time of exposure.
- The test coating is designed for use in environmental monitoring or personal dosimetry. It is simple in structure and easily manufactured at reasonable cost. The coating which may be provided in the form of a tape or sheet incorporates all of the reagents needed for the detection and quantification of the gas and requires no processing to provide a permanent record. It may be exposed to an environment for a fixed period of time thus determining the total exposure to the gas in question over the period, e.g. an 8 hour working shift. Quantitative analysis may be accomplished by use of conventional spectrophotometers or densitometers or, less accurately, by comparison with colour standards.
- The support used in the coating may be of any suitable material known for such use in photography or other similar arts. Examples of such supports include glass, paper, resin coated paper, polymeric films such as cellulose

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lose acetate, polycarbonates or polyesters, and metal. The support should be inert to substances in the reagent layer. In a preferred embodiment, the support used is impermeable to the gas under analysis.

5 In order to prevent premature reaction, the coating made on a gas-impermeable support is covered with a temporary gas-impermeable cover sheet. Self-adhesive tapes of gas-impermeable materials may be used, e.g. polyvinyl chloride or aromatic polyester self-adhesive tapes.

10 The reflection density of the sensitive layer which becomes coloured on exposure to the gas may be ascertained when opaque supports are used. In a preferred embodiment the support employed is capable of transmitting at least certain wavelength bands of electromagnetic radiation in the range of between 200 and 900 nm.

15 The reagent layer contains the reagents intended to undergo colour-forming or other radiation detectable reactions with the gas in order that reaction product concentrations per unit area can be quickly and readily determined. For the purposes of this discussion, the reagent layer specified will be that used for the determination of ethanol. Other gases may be determined by similar methods by variation in the basic reagent system components used and by any required modification in the conditions of preparation.

20 The quantification of ethanol by reaction of ethanol with dichromate in the presence of a concentrated acid, particularly sulphuric acid, to produce a coloured product is well known. Useful dichromates are available commercially. Concentrated aqueous solutions of suitable dichromates such as saturated solutions of sodium or potassium dichromate are used to prepare coatings as described herein.

25 Organic or inorganic acids such as sulphuric or phosphoric acids may be used as the concentrated acid. In order to improve physical characteristics such as dryness, durability and hardness as well as handling safety, the acid is adsorbed onto the adsorbent to provide a stiff paste containing the acid. The particles containing the adsorbed acid may then be incorporated in the binder in a conventional fashion before coating the layer. The coatings are not significantly hygroscopic as opposed to unadsorbed concentration sulphuric acid which is. A final coated weight of sulphuric acid of up to 45% is attainable and acceptable for the preparation of useful coatings. The weight ratio of acid to adsorbent may be regulated by varying the particle size and hence surface area of the latter. Equal weights of acid and adsorbent will, however, often prove convenient.

65 The gas sensitive layer may be coated on

the support by forming suitable coating compositions, coating them onto the support and then drying the layer. The coating composition may contain many other additives, for example, surfactants, lubricants, plasticizers and the like which aid in the coating process or improve the characteristics of the final coating but do not interact harmfully with the assay reagents.

Typically, the present ethanol-sensitive coatings are prepared as a layer about 50 μ thick containing from 0.12 to 1.7 g of sodium dichromate from a saturated solution and from 1 to 13 g of $\text{SiO}_2/\text{H}_2\text{SO}_4$ in up to 17 g of 10% aqueous polyacrylamide on a support. Concentrations of polyacrylamide below 8% might present drying problems; concentrations above 12% may present dissolution and viscosity problems. Typical drying times for the coated layer are less than one hour at room temperature and humidity.

The coating undergoes reaction upon exposure to the atmosphere in 4-5 days. The keeping time may be extended to 3-4 weeks by excluding air from the reactive surface with a relatively alcohol-impermeable covering, for example a poly(ethylene terephthalate) and/or polyvinyl chloride self-adhesive tape.

As prepared herein, the coating may be used to detect ethanol vapours produced from 0.3% ethanol, being equivalent to the approximate concentration of ethanol in breath at 80 mg/100 ml blood level. In addition, the coating may be directly spotted with a suspected ethanol solution to produce a colour change.

The description is further illustrated by the following example:

Example

A coating containing all the necessary reagents for the analysis of reducing gases was prepared in the following manner: Silica gel (Silica Gel for Chromatography, 100-200 mesh) was soaked in concentrated H_2SO_4 . The excess, unadsorbed acid was removed by filtration through a sintered glass funnel under vacuum. A coating mixture containing polyacrylamide 100 (American Cyanamid Company) (3 ml, 10% aqueous solution), sodium dichromate (1 ml, saturated aqueous solution), and the acidified silica gel (1 g.) was coated on a poly(ethylene terephthalate) support and dried for an hour at room temperature.

To evaluate the coating a 5% ethanol solution was spotted on the sample. The coating turned green within one minute. When sealed in a polythene bag with 0.3% ethanol solution, which produces the approximate concentration of ethanol in breath at 80 mg/100 ml. blood level, the layer turned green within about 20 minutes.

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WHAT WE CLAIM IS:-

1. A chemical monitoring device containing a concentrated acid which comprises a support having coated thereon a layer comprising a concentrated acid adsorbed onto particles of an inert adsorbent, a colour-forming reagent and a binder comprising a non-degradable synthetic polymer.
2. A device as claimed in claim 1 in which the particles of inert adsorbent have an average size of from 1 to 100 microns.
3. A device as claimed in claim 2 in which the particles have an average size of from 3 to 30 microns.
4. A device as claimed in any of claims 1 to 3 in which the inert adsorbent is alumina, Kieselguhr, titanium dioxide or silica gel.
5. A device as claimed in any of claims 1 to 4 in which the concentrated acid is concentrated sulphuric, nitric or phosphoric acid, glacial acetic acid or saturated aqueous citric acid.
6. A device as claimed in any of claims 1 to 5 in which the colour-forming reagent is a water soluble dichromate.
7. A device as claimed in any of claims 1 to 6 in which the binder is a polyacrylamide homopolymer or copolymer.
8. A device as claimed in any of claims 1 to 7 in which the support is impermeable to the gas under analysis.
9. A device as claimed in claim 8 in which the coated layer is covered with a temporary gas impermeable cover sheet.
10. A chemical monitoring device substantially as described herein and with reference to the Example.

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